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AN ELECTROLYTIC METHOD OF PREVENTING CORROSION OF IRON AND STEEL

BY

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Original from PRINCETON UNIVERSITY

AN ELECTROLYTIC METHOD OF PREVENTING CORROSION OF IRON AND STEEL.

By J. K. Clement and L. V. Walker.

INTRODUCTION.

In the hope of developing means for reducing the losses resulting from the corrosion of iron and steel used in mine equipment, the Bureau of Mines has undertaken a thorough investigation of the problem. This preliminary report is an account of experiments made to develop an electrolytic method for preventing corrosion. The results stated appear to justify the hope that the method may be applied successfully in mines for protecting iron and steel against the corrosive action of acid underground waters, as well as for protecting structures and machinery that are exposed to the acid waters of streams.

ELECTROLYTIC THEORY OF CORROSION.

According to the electrolytic theory of corrosion, when iron is oxidized in the wet way it first goes into solution as ferrous ions. The ferrous ions are then oxidized by the oxygen present in the water to ferric ions and are precipitated as ferric hydroxide. Simultaneously with the formation of ferrous ions hydrogen is liberated on the surface of the iron. The passage of the iron from the atomic to the ionic state and the passage of the hydrogen from the ionic to the atomic state are accompanied by a transfer of electricity. The ferrous ions derive from the metal surface a charge of positive electricity, the hydrogen ions give up a charge of positive electricity and a current flows through the metal from the point where hydrogen is liberated to the point where iron is dissolved.

If iron and a metal having a greater "solution tension" than iron—for example, zinc—be immersed in an acid solution and connected externally through a wire, zinc will go into solution, hydrogen ions will be liberated on the iron, and current will flow through the wire from iron to zinc. The potential difference between zinc and iron, being opposed to the potential difference between iron and electrolyte, neutralizes the force required to pull the positively charged iron ions away from the negatively charged surface of the iron electrode. Consequently iron does not go into solution.

In place of zinc other conducting materials—for example, carbon—may be used as the anode, and the electromotive force required may be furnished from an external source. In this way electric energy derived from either a battery or a generator may be used to protect metals from corrosion under water.

A vast amount of work, both theoretical and experimental, has been directed to the study of the causes of corrosion and the development of methods for its prevention, and the literature on the subject is extensive.[a]

Very little attention, however, seems to have been given to the development of an electrolytic method for the prevention of corrosion. An electrolytic method used to some extent for the protection of boilers[b] consists in submerging a bar of zinc in the boiler water and connecting the zinc electrically with the boiler plates. After the experiments described in this paper were begun it was found that an Australian inventor had patented an electrolytic process for the protection of metals.[c] The writers have been unable to find any data published by the inventor.

In a paper presented before the Sydney (Australia) section of the Society of Chemical Industry, Harker[d] describes experiments on the prevention of the corrosion of iron by electrolysis. He determined the current density necessary to prevent the corrosion of steel plates submerged in $\frac{N}{25}$ H_2SO_4, tap water, sea water, and "dilute acid." The results of Harker's experiments, which furnish the only data on the subject that the writers have been able to find, will be discussed in connection with the results of their own experiments.

EXPERIMENTS BY STANTON.

Experiments to devise an electrical method for preventing the corrosion of steel immersed in acid water were undertaken at the Pittsburgh experiment station of the Bureau of Mines in May, 1911, by F. M. Stanton, chemist.

Stanton's work included laboratory experiments and tests with plates immersed in Monongahela River. In the laboratory experiments steel plates measuring 6 mm. by 19 mm. by 51 mm. were suspended in separate vessels in $\frac{N}{5}$ H_2SO_4. In one vessel a carbon rod was suspended near the steel plate and connected to the positive pole of a storage battery, the negative pole of which was connected with the steel plate. In the second vessel the steel plate was not protected. After 24 hours' exposure the unprotected plate lost in weight 1.81 grams and the protected plate only 0.0012 gram.

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[a] Cushman, A. S., and Gardner, H. A., Corrosion and Preservation of Iron and Steel, 1910, 373 pp.
Friend, J. Newton, The Corrosion of Iron and Steel, 1911, 300 pp.
List of references to books and magazine articles on metal corrosion and protection, reprinted from the Monthly Bulletin, Carnegie Library of Pittsburg, July, 1909.

[b] Paul, J. H., Corrosion in steam boilers. Trans. Soc. Eng., vol. 31, 1891, p. 147.

[c] Preventing corrosion of metals by means of electricity. Elec. Review and West. Electn., vol. 58, 1911, p. 326.

[d] Harker, G., and McNamara, J., Electrolysis as a means of preventing the corrosion of iron and steel. Jour. Soc. Chem. Ind., vol. 29, 1911, p. 1286.

In the tests with plates submerged in Monongahela River a much smaller current density was used than in the laboratory experiment, and the protection against corrosion was much less complete. The plates used in these tests measured 6 by 8 inches by ⅛ inch. The protecting current was furnished by a 2-volt accumulator. The loss in weight of the two plates at the end of 15 days' immersion was 0.7 gram for the protected plate and 10.9 grams for the unprotected plate.

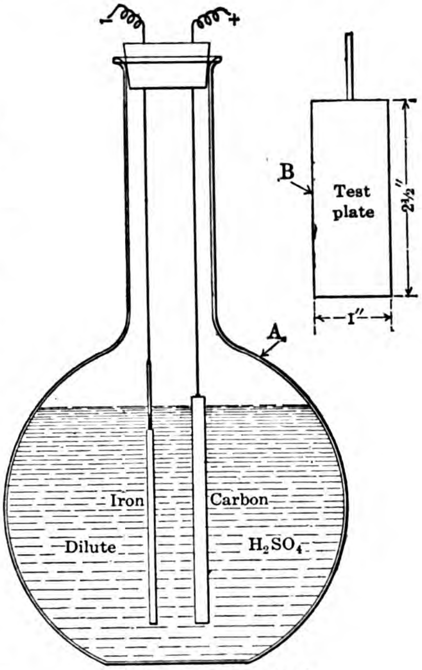

FIGURE 1.—Apparatus 1.

EXPERIMENTS TO DETERMINE CURRENT DENSITY REQUIRED TO PREVENT CORROSION.

Mr. Stanton left the service of the Bureau of Mines in July, 1911, and the work was interrupted until December, 1911, when experiments were resumed by the authors and A. E. Hall.

The object of these experiments was to determine the current density required to prevent, under various conditions, the corrosion of steel plates in acid water.

EXPERIMENTS WITH APPARATUS 1.

The first method of determining the protective effect of small currents was suspending an iron plate (dimensions shown in fig. 1) in an 800-c. c. flask parallel to a ¼-inch carbon rod and ½ inch from it. With storage batteries as a source of energy, a current was passed from the carbon rod through the dilute sulphuric acid in the flask to the iron plate. The current strength was regulated by the use of variable resistances and the current was measured by the drop in potential over a known resistance by means of a Siemens & Halske millivolt meter. Electrical connections were always completed before any acid was put in the flask and the current was regulated immediately thereafter. In order to save time six such flasks were used in each experiment.

The results of the first series of experiments are shown graphically in figures 2 and 3. The experiments represented by the curves in figure 3 were made under uniform conditions, except that the duration of the test was varied. These experiments indicate that a certain definite current density must be maintained in order to reduce the corrosion loss to a minimum. On account of the poor agreement between the results of individual experiments a second type of apparatus was designed.

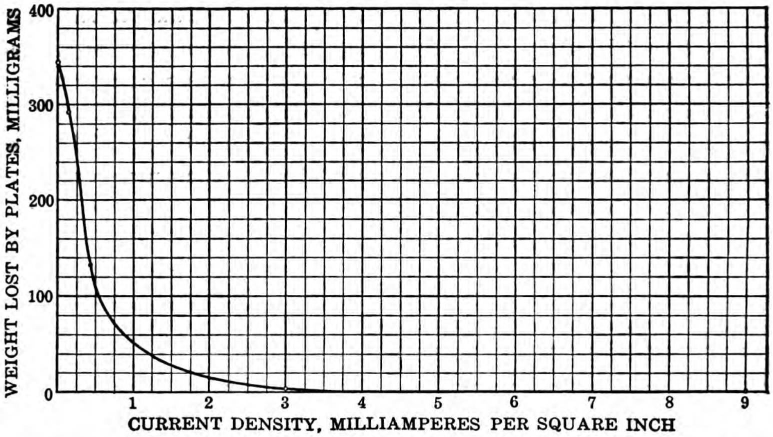

FIGURE 2.—Relation between weight lost by plates and current density in test with apparatus 1. Length of test, 45 hours; concentration of acid, $\frac{N}{5}$; electrolyte not stirred.

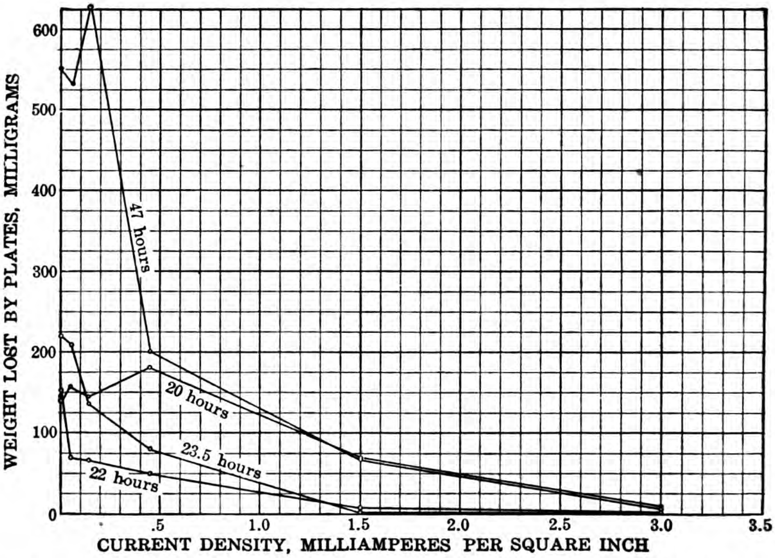

FIGURE 3.—Relation between weight lost by plates and current density in test with apparatus 1. Concentration of acid, $\frac{N}{10}$; electrolyte not stirred.

EXPERIMENTS WITH APPARATUS 2.

In the experiments with this apparatus, in order to eliminate as far as possible any error due to differences in acid concentration about the iron plates, all the test plates were immersed in a common vessel. Another improvement was the employment of an acid reservoir, and a constant-level device by means of which fresh acid could be fed, at any desired speed, into the containing vessel and the excess electrolyte could be siphoned off through the level bottle into the overflow. The vessel was a porcelain-lined cylindrical dish having a capacity of about 2,250 c.c. and the plates were supported by a thin wooden cover at points on a circle 4 inches in diameter, the carbon-rod anode being suspended from the center. The plates were set equidistant from each other and, of course, from the anode. In the six experiments conducted according to this scheme the acidity of the electrolyte was determined at the end of each run. There was always a considerable decrease in acid strength which could be eliminated to a large degree by increasing the rate at which the acid was fed. For example, at the end of a 27-hour run, started with $\frac{N}{10}$ H_2SO_4, the acidity was $\frac{85}{1000}$ normal. As all the plates, however, were subjected to the same variation in the strength of the electrolyte, the loss in weight of the plates should be comparable.

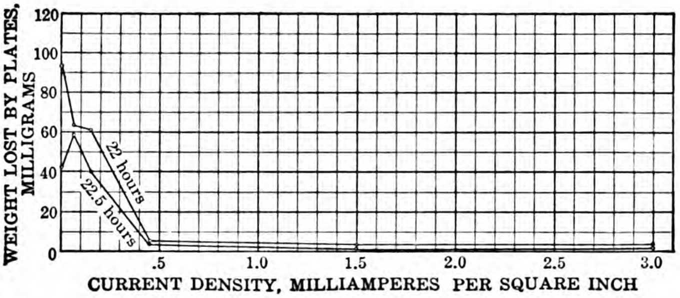

FIGURE 4.—Relation between weight lost by plates and current density in tests with apparatus 2. Concentration of acid, $\frac{N}{10}$; electrolyte not stirred.

The curves in figure 4 represent the plate losses during experiments 11 and 12, in which apparatus 2 was used. Though the results do not check exactly, they are more consistent than those of the previous experiments. The indications from the experiments of series 1 and 2 are that a current of 0.4 milliampere to the square inch (figuring one-third of the back of the plate as is customary) will prevent 93 to 94 per cent of the corrosive action of $\frac{N}{10}$ H_2SO_4 when the solution is not stirred.

EXPERIMENTS WITH APPARATUS 3.

Apparatus 3, the one finally adopted, is shown in figure 5. It differs from apparatus 2 chiefly in having a greater capacity, 4½ liters, and in being provided with an attachment for stirring the electrolyte. The plates were arranged as before, except that they were set 2½ inches from the carbon rod, instead of 2 inches, in order to keep them under water when the electrolyte was rotating rapidly. For stirring, a low-power hot-air engine gave fairly constant speed and could be run overnight. In order to be able to calculate the current densities with greater accuracy, all surfaces of the plates except the one facing the anode were protected by being painted with bakelite lacquer. An application of four or five coats gave a good, smooth surface that was impenetrable by acids.

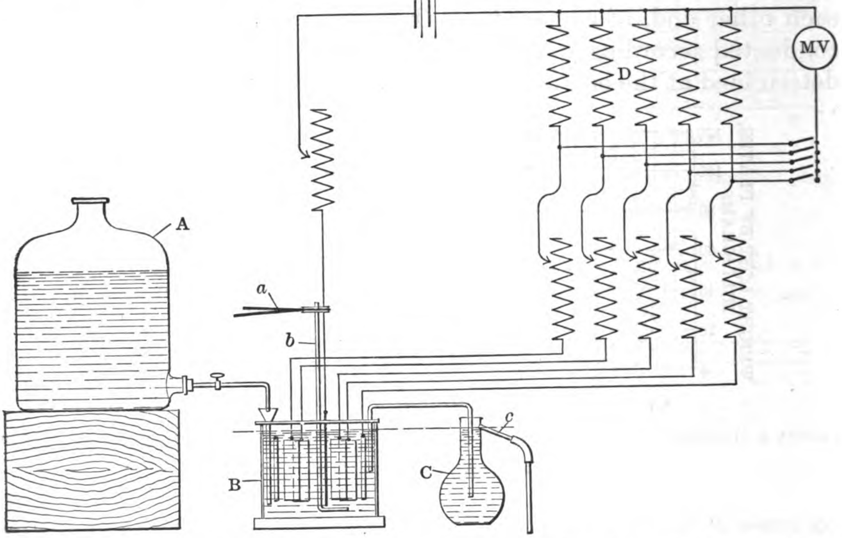

FIGURE 5.—Apparatus 3. A, acid reservoir; B, electrolyte tank; C, level bottle; D, known resistance; *a*, belt to motor; *b*, stirrer; *c*, overflow.

The first few experiments using the final apparatus were run with the stirrer rotating at 660 revolutions per minute, and under these conditions currents that had protected the iron plates almost completely in still water were absolutely useless. On reducing the stirring rate to 35 revolutions per minute in $\frac{N}{100}$ acid, small currents were again effective, and runs could be checked with a fair degree of accuracy.

Figure 6 shows the results of experiments using $\frac{N}{100}$ H_2SO_4 as electrolyte and with the stirrer running at 35 to 40 revolutions per minute. At frequent intervals in this series of experiments the electrodes were removed and weighed. The curves in figure 6 show that the plates having a current density of 0.4 milliampere per square inch or less continued to lose weight during the entire period of the experiments. Plates protected by current densities greater than 0.4 milliampere to the square inch exhibited a marked initial corrosion and thereafter no further loss in weight. The initial loss in weight of the plates protected by current densities greater than 0.4 milliampere is probably due to the fact that at the start of the experiment the plates were covered by a film of air the oxygen of which acted as a depolarizer. Some time elapsed, therefore, before the plates became completely polarized, and during this period they were incompletely protected by the current. The difference between the rate of loss in weight of the unprotected plate during the initial period and the rate of loss during the remainder of the experiment is probably due to a difference between the structure of the original surface of the metal and the interior, as well as to the action of the film of air surrounding the plate at the beginning of the experiment.

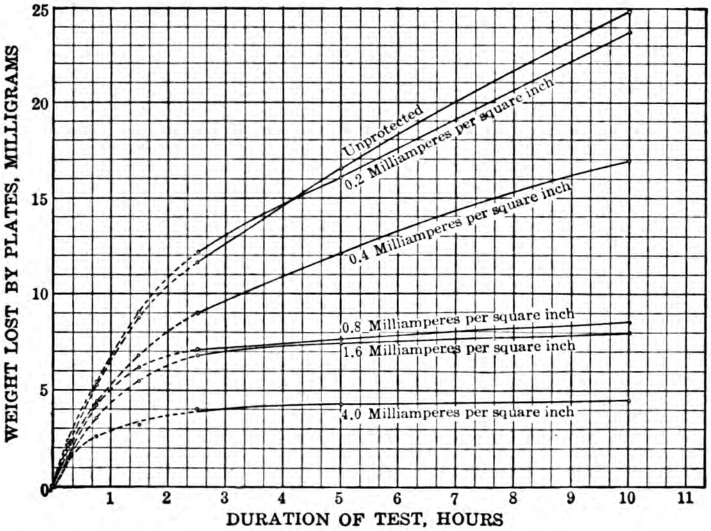

Figure 6.—Effect of current density on relation between weight lost by plates and duration of test. Apparatus 3 used; concentration of acid, $\frac{N}{100}$; speed of stirrer, 35 revolutions per minute.

EFFECT OF PASSING OXYGEN THROUGH ELECTROLYTE.

On account of the depolarizing effect of oxygen it was to be expected that the amount of oxygen present in the electrolyte would be an important factor in determining the rate of corrosion. Figure 7 shows the results of a series of four experiments made to ascertain the effect of increasing the quantity of oxygen in the electrolyte. The composition of the electrolyte, $\frac{N}{100}$ H_2SO_4, and the speed of the stirrer, 35 revolutions per minute, were the same in all four experiments. The lower curves represent two experiments with the usual proportion of oxygen present. The electrolyte was prepared by adding sulphuric acid to distilled water without any special precautions to exclude the air or to aerate the solution. In experiments 38 and 39, represented by the two upper curves of figure 7, the electrolyte was saturated with oxygen by rapidly bubbling oxygen through it during the experiment. The curves show that increasing the amount of oxygen in the solution decidedly accelerated the rate of corrosion, the loss of weight in 24 hours being about twice as great when oxygen was passed through the electrolyte as when oxygen was supplied by diffusion only. It follows from this that the rate of corrosion in any acid solution depends on the degree of aeration of the solution.

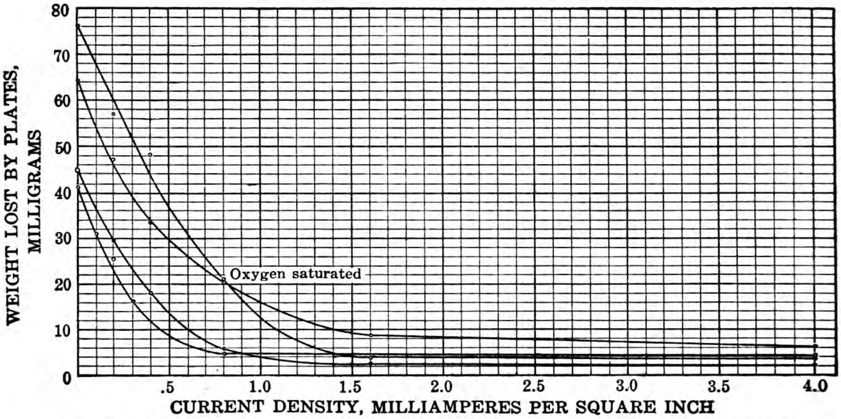

FIGURE 7.—Effect of oxygen on relation between weight lost by plates and current density in test with apparatus 3. Duration of test, 24 hours; concentration of acid, $\frac{N}{100}$; speed of stirrer, 35 revolutions per minute.

EFFECT OF STIRRING.

The enormous acceleration of corrosion produced by extremely rapid stirring, 660 revolutions per minute, has already been noted (p. 10). On account of the magnitude of the effect, a series of experiments was made in which the speed of the stirrer was the only variable. The results are shown in figure 8. An examination of the curves shows that, the other conditions being constant, the loss in weight by corrosion varies directly as the rate of stirring. The increase in the speed of corrosion with the increase in the rate of stirring is further illustrated by figure 9, in which the loss in weight of the plates for several different current densities is plotted against the rate of stirring.

These results demonstrate that the rate of flow of acid solution over the metal surface is an important factor in determining both the amount of corrosion and the current density necessary to protect the metal. The decided increase in the speed of corrosion produced by circulation of the electrolyte is not surprising, since the flow of solution over the metal not only hinders the exhaustion of hydrogen ions in the vicinity of the anode but provides a continual supply of oxygen and tends to destroy any film of hydrogen that may form on the surface of the cathode.

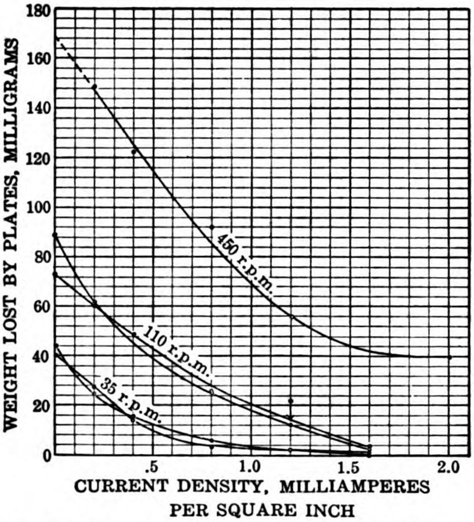

FIGURE 8.—Effect of speed of stirrer on relation between weight lost by plates and current density in test with apparatus 3. Duration of test, 24 hours; concentration of acid, $\frac{N}{1000}$.

ACID CONCENTRATION.

That the rate of corrosion of iron or steel in sulphuric acid solutions increases with the concentration of the acid is well known. Though the acid concentration in the waters of nearly all streams in which iron is being corroded is much less than $\frac{N}{100}$, the concentration used in most of the experiments described, the application of the method

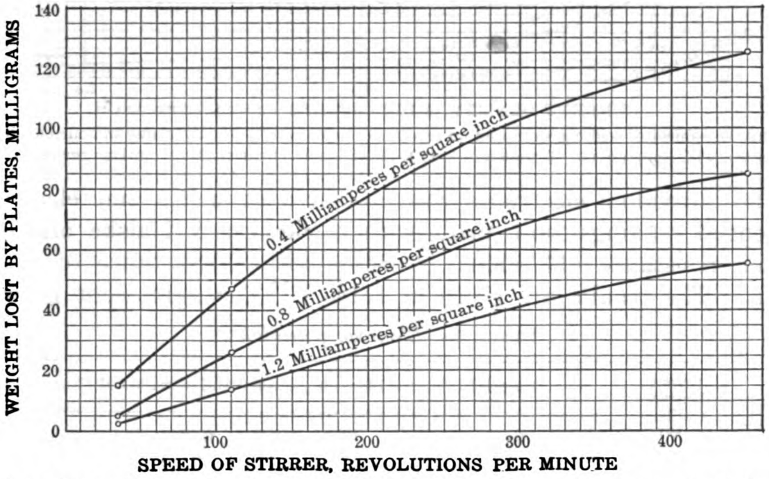

FIGURE 9.—Effect of current density on relation between weight lost by plates and speed of stirrer in test with apparatus 3. Duration of test, 24 hours; concentration of acid, $\frac{N}{1000}$.

to more concentrated solutions seemed of sufficient interest to justify experiments with stronger solutions. Accordingly a series of experiments was made in which the concentration of sulphuric acid was varied from $\frac{N}{1000}$ to $\frac{N}{10}$. The results are represented by the curves in figure 10. They indicate that, although a considerably greater current density would be required, the method would be effectual even in $\frac{N}{10}$ H_2SO_4. The curves for experiments with $\frac{N}{100}$ and $\frac{N}{1000}$ acid are practically identical, showing that between these limits of acid concentration there is no change in the rate of corrosion greater than the limit of error of observation. This result, though unexpected, was confirmed by repeated experiments.

POTENTIAL OF IRON ELECTRODES.

In the majority of the experiments, in addition to the current flowing into the different electrodes being measured, the potential of the individual electrodes was measured against a zinc electrode. Potential readings were made with a Siemens & Halske millivoltmeter, resistance 450 ohms, in series with a resistance of 17,550 ohms. The results are given in Table 1. The agreement between

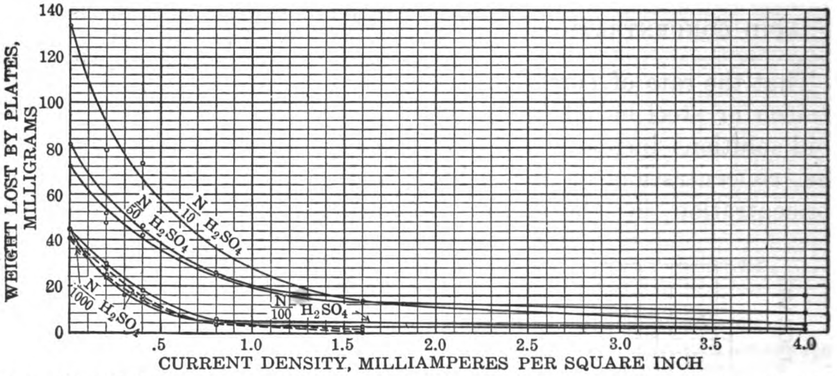

Figure 10.—Effect of acid concentration on relation between weight lost by plates and current density in test with apparatus 3. Duration of test, 24 hours; speed of stirrer, 35 revolutions per minute.

Table 1.—*Electrode potentials.*

Current (milliamperes).	Current density (milliamperes per square inch).	Potential difference between protected and unprotected plate.								
		Exp. 30	Exp. 31	Exp. 32	Exp. 35	Exp. 36	Exp. 37	Exp. 38	Exp. 39	Exp. 40
0.50	0.2	0.021	0.004	−0.021	−0.024	0.004	0.012	0.027	−0.005	−0.004
1.00	.4	....	− .003	− .012	.003	.022	.008	− .018	− .051	− .024
2.00	.8	.198	.025	.102	.078	.073	.044	.012	.000	.015
4.00	1.6	.433	.114	.237	.186	.187	.108	.102	.081	.090
10.00	4.0	.723	.289	.429	.372	.392	.304	.315	.295	.303

Rate of stirring, 35 revolutions per minute. Concentration of acid, $\frac{N}{100}$ H_2SO_4.

the potentials of electrodes having the same current density is only fair. In $\frac{N}{100}$ H_2SO_4, and with a rate of stirring of 35 revolutions per minute, a current density of 0.8 milliampere per square inch generally produced an increase in the potential of the iron electrode of about 0.04 volt. In the table, potential differences are positive when the potential of the protected plate is positive to the unprotected plate; that is, when the potential difference between the zinc electrode and the protected plate is decreased by the protecting current. As small an increase as 0.04 volt in the potential of the test plates sufficed, therefore, to protect them against corrosion. This effect is too small, in view of the variations found between the results of different experiments made under like conditions of acid concentration and rate of stirring, to be of value as a measure of the degree of protection afforded by the current against corrosion.

CURRENT DENSITY.

In the experiments that have been described, the following factors were found to influence the current density required to protect the corrosion of steel plates submerged in sulphuric acid solutions:

Rate of stirring of electrolyte.
Proportion of free oxygen present.
Acid concentration.

Of these the last named is probably least important, especially in dilute solutions.

The rate of stirring of the electrolyte is by far the most important factor. Under normal conditions the supply of oxygen in the immediate vicinity of submerged metal surfaces depends largely on the rate of flow of the solution over the surfaces. With the lowest rate of stirring used in the experiments, 35 revolutions per minute, and in acid concentrations not greater than $\frac{N}{100}$, a current density of from 0.5 to 0.8 milliampere reduced the corrosion loss to a negligible quantity, whereas with the stirrer rotating at a speed of 450 revolutions per minute and a current density of 2 milliamperes the corrosion loss amounted to 25 per cent of the loss on the unprotected plate.

The electromotive force required to maintain a given current density depends on the kind of anode used, on the position of the anode with respect to the surface to be protected, and on the electrical conductivity of the water. In tests now being made on steel plates submerged in the Monongahela River, at Braddock, Pa., with a distance of about 1 foot between electrodes, an electromotive force of 5 volts is used. Decreasing the distance between electrodes would permit the use of a lower voltage.

If 1 milliampere per square inch (6.45 milliamperes per square centimeter) be taken as the current density required to prevent corrosion under a given set of conditions, then, with an electromotive force of 5 volts, the power consumption per square foot of protected surface will be approximately 0.75 watt, and the electric energy will be:

$$\frac{0.75\times24\times365}{1,000}=6.6 \text{ kilowatt-hours per square foot per year}$$

or 71 kilowatt-hours per square meter of surface per year.

HARKER'S EXPERIMENTS.

Harker, from his experiments (p. 6), concluded that in employing the electrolytic process the current required to prevent corrosion might be calculated from the rate of corrosion of iron or steel under the given conditions and the electrochemical equivalent of iron. This conclusion he confirmed by one set of experiments in which, however, he used iron anodes. In experiments with a platinum anode, Harker found that the protection afforded by the current to the iron cathode was somewhat less than when an iron anode was used. He concludes that "the electrolytic process does not actually prevent metal from entering the solution from the cathode, for where the rate remains constant deposition is taking place from the anode, yet the tendency of the metal to pass into solution is much diminished."

APPLICATION OF HARKER'S RULE.

The authors in all of their experiments used carbon anodes. Harker's method of calculating the current density required to prevent corrosion was based on experiments with an iron anode. If, then, as Harker suggests, the protective action of the current is largely due to the transfer of iron from the anode to the cathode it should not be expected that his method would hold for experiments with a carbon anode.

The method offers, however, such a simple procedure for arriving at the proper current density required to prevent corrosion that it seemed worth while to apply it to the results of the authors' experiments. In Table 2 are given the results of these experiments, to which have been added the values for the current densities calculated from the loss in weight of the unprotected plates and the electrochemical equivalent of iron. The "observed" values for the current densities in column 6 were obtained graphically from the curves in figures 2, 3, 4, 7, 8, and 10. The agreement between the observed and calculated values is remarkably good. The differences are well within the experimental error.

It may therefore be concluded that Harker's method of calculating the current density required to prevent corrosion, from the loss in weight of the metal under the given conditions, is not limited to cases in which an anode of the same metal is used.

TABLE 2.—*Results of experiments to determine current density necessary to prevent corrosion.*

Results plotted in figure—	Number of experiment.	Acid concentration.	Rate of stirrer (r. p. m.).	Length of run (hours).	Observed current density (milliamp. per sq. in.).	Calculated current density (milliamp. per sq. in.).	Weight lost by unprotected plate (mg.).	Type of apparatus used.
2	4	$\frac{1}{8}$ N.....	....	45	3.0	2.2	344.6	1
3	6	$\frac{1}{10}$ N....	....	47	3.0	3.4	551.5	
3	7	...do....	....	20	3.0	2.0	140.5	
3	9	...do....	....	23.5	1.5	2.7	221.0	
3	8	...do....	....	22	1.5	2.0	151.5	
4	11	...do....	....	22.5	.45	.5	43.0	2
4	12	...do....	....	22	.45	1.2	93.5	
7	39	$\frac{1}{100}$ N [a]..	35	24	1.6	1.3	75.9	3
7	38	...do [a]...	35	24	1.6	1.0	64.3	
7	40	$\frac{1}{100}$ N...	35	24	.8	.7	44.6	
7	30	...do....	35	24	.8	.7	41.4	
8	48	$\frac{1}{1000}$ N..	450	24	2.0	2.7	170.0	
8	45	...do....	110	24	1.6	1.4	88.3	
8	46	...do....	110	24	1.6	1.2	72.8	
8	42	...do....	35	24	.8	.7	44.5	
8	41	...do....	35	24	.8	.6	41.0	
10	51	$\frac{1}{10}$ N....	35	24	1.6	2.2	133.8	
10	49	$\frac{1}{50}$ N....	35	24	1.2	1.1	71.3	
10	50	...do....	35	24	1.2	1.3	80.9	

[a] Oxygen saturated.

THE NATURE OF THE ACTION OF THE ELECTRIC CURRENT.

Harker's view that the current does not actually prevent metal of the cathode from entering the solution, and that its protective action is due to the deposition of metal from the anode, is contradicted by the authors' experiments in which carbon anodes only were used. The action of the current, then, is to prevent the formation of ferrous ions at the cathode by increasing its negative charge. In the absence of any depolarizer, dissolved oxygen especially, an infinitesimal current should produce an electromotive force large enough to neutralize the solution tension of the metal. When oxygen is present it combines with the hydrogen ions liberated at the cathode and hinders polarization. The supply of hydrogen ions furnished by the current must be equivalent to the supply of oxygen in the vicinity of the cathode before the solution tension of the cathode can be completely neutralized.

CONCLUSION.

It has been shown that the corrosion of iron submerged in sulphuric acid solutions may be prevented by imposing a counter electromotive force. The density of the current required to prevent corrosion depends on various factors, the more important being: Acid concentration, amount of dissolved oxygen, and rate of circulation of the electrolyte. The influence of these factors has been studied and curves have been given showing the current density necessary under various conditions. It has been found that the current density required can be calculated, within the limit of experimental error, from the loss in weight of the unprotected metal under the given conditions.

PUBLICATIONS ON MINE ACCIDENTS AND METHODS OF MINING.

The following Bureau of Mines publications may be obtained free by applying to the Director, Bureau of Mines, Washington, D. C.:

BULLETIN 10. The use of permissible explosives, by J. J. Rutledge and Clarence Hall. 1912. 34 pp., 5 pls., 4 figs.

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